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## PAPERS.

## IODOFORM.

*Preparation from Acetone and Hypochlorites; also, a Trial by Electricity.*

BY S. R. BOYCE, LAWRENCE.

As everyone knows, the expensive part of iodoform is the iodine used in its manufacture, and as this constitutes by weight 96.69 per cent. of the finished product, it will readily be seen that its production must always be associated with expense. In working on a new formula for its production, the difficulty we aim to overcome is the appropriation of iodine to form iodides which are lost in or have to be recovered from the mother liquor.

The best yields I have been able to find on record from any of the old methods were recorded by Hager, who used Filhol's process and claimed 72 per cent. of the iodine appropriated; but Wilder, following the same directions, was able to obtain only 42 to 53 per cent. (Proc. Am. Phar. Ass. 1875, 717.)

We are now prepared to formulate a method by which practically all the iodine used is converted into iodoform and the reaction is completed at once without the labor and inconvenience of decomposing the mother liquor with nitric acid, as in Filhol's process.

The reaction is accomplished by the action of an alkaline solution of hypochlorite on potassium iodide in presence of acetone. The chlorine and iodine exchange positions, forming chloride and hypo-iodite. The iodine of the hypo-iodite immediately unites with a portion of the methyl in the acetone, and the solution becomes turbid from the abundant precipitation of iodoform. The greater part of the acetone appears to be appropriated to the formation of acetates and carbonates of the alkalies present. The following equation would probably approximate the reaction:  $3 \text{ KI} + 3 \text{ KClO} + \text{C}_3\text{H}_6\text{O} = \text{CHI}_3 + 3 \text{ KCl} + \text{KC}_2\text{H}_3\text{O}_2 + 2 \text{ KOH}$ .

The operation requires no heat, and the precipitation is best conducted in a tall cylindrical vessel, allowing the solution of hypochlorite to fall into the other solution drop by drop as long as a precipitate is formed. The vessel should be large, as it requires about three parts of the former solution to one of the latter. As it is difficult to decant the hypo. solution perfectly clear, I prefer to place a filter over the receiver and turn on a small amount at a time, allowing it to drop as it comes through the filter. The constant dropping keeps the solution in motion; and in addition to this the receiver should be given an occasional rotary motion to facilitate the agglomeration and precipitation of the very minute crystals formed.

A complete precipitation can be accomplished in an hour; and if any considerable length of time is occupied, the solution should be protected from the light by wrapping a piece of paper around the container, or by other simple device.

The solutions are made as follows,\* and special attention should be given to the amount of acetone used, since experiments proved that smaller amounts would give proportionately smaller results:

## SOLUTION No. 1.

Potassium iodide.....	gm. 50
Caustic soda.....	gm. 5
Acetone .....	gm. 40

Dissolve the KI and the NaOH in one litre of water, and add the acetone.

\*Modification of a formula given by Sulliot and Raynand, *Chem. Zeit.*, Jan. 16, 1889. Also, *Bull. Soc. Chem.* 1889, 1, 3.

Solution No. 2 is the official Liquor Sodæ Chloratæ of the U. S. P., and should be added to No. 1 in small portions, as stated above. In preparing this solution, add more sodium carbonate if the calcium is not all precipitated.

In repeated operations I obtained 89 to 91 per cent. of the hypothetical amount of iodoform, which is equivalent to the same percentage of the iodine employed. The mother liquor contains 2 per cent. of the iodine as iodides, which leaves about 8 per cent. to be accounted for. We think by proper adjustment of the solutions the above 2 per cent. can be recovered as iodoform, and we are also of the opinion that the 8 per cent. mentioned exists as iodoform dissolved in the mother liquor.

*Electricity.*—Seeing statements to the effect that iodoform could be produced by electricalizing solutions of the iodides with acetone or aldehyde receiving a continuous stream of  $\text{CO}_2$ , and the accompanying statement that if sodium or calcium iodide was used, the  $\text{CO}_2$  was unnecessary. I thought I might be able to take a step toward bringing pharmaceutical preparations into communion with the electric age in which we live. I tried the experiment, and it was a *grand success*. With five Bunsen cells arranged in series, I was able to produce iodoform from all the different substances I tried containing iodine either free or in combination as soluble iodides. The precipitate formed slowly and steadily from all but the calcium iodide, from which it was thrown down very rapidly. But on examining the product, I was tempted to call it something like *iodocalioform* to indicate its true composition, containing 25 per cent. of calcium carbonate.

Electrolysis of calcium iodide might be a profitable method for the preparation of iodoform, but since I tried it, I maintain that this method would be more consistent with the operations of the Physics Department than those of the Pharmaceutical.

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## ESTIMATION OF VOLATILE OIL IN MUSTARD.

BY L. E. SAYRE, LAWRENCE.

The object of this investigation has been to obtain, if possible, a simple method by which to estimate the value of mustard. The elaborate analyses recommended by different chemists, by which the various proximate principles of mustard are isolated and weighed, take considerable time, and admit of many sources of error. Hassell's method of estimating the volatile oil, by receiving the distillate from the aqueous mixture of mustard, in ammonia, evaporating, and weighing the crystals of thiosinamin, is apt to give variable results, because of the fact that the volatile oil escapes, to a greater or less extent, through the ammonia, in spite of the greatest care on the part of the operator.

The important constituents of mustard, it may be known, are *sinigrin* (myronate of potassium), from the black and *sinalbin* from the white mustard. Besides these they contain *myrosin*, which acts as an albuminous ferment. When powdered mustard is moistened this ferment acts upon the glucosides, forming volatile oil, sulphate of potassium and glucose as products of decomposition, or fermentation.

A favorite method of estimating mustard, by some chemists, has been to convert the whole of the sulphur contained in the mustard into sulphates by the action of nitric acid, and to estimate the sulphate by use of barium chloride, deducting from the total sulphates the amount of inorganic sulphates found in the ash.

It occurred to me that there might be a means by which mustard might be estimated by distillation of the volatile oil into some solution containing a substance which would unite with the sulphur or compound of sulphur so readily that none of